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A STUDY OF SOME CERAMIC BODIES OF LOW ABSORPTION MATURING AT TEMPERATURES BELOW 1,000° C.

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ABSTRACT

This paper contains a report of an investigation involving the development and testing of a number of ceramic bodies of the whiteware type containing synthetic flux and maturing below 1,000° C. The bodies compare favorably in transverse breaking strength with earthenware, are nearly white in color and vary in absorption from 1 to 5 per cent. The resistance to mechanical abrasion is higher than that of nonceramic flooring materials, such as marble. Glazed specimens resisted crazing satisfactorily in laboratory tests.

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I. INTRODUCTION

The idea of using artificially prepared fluxing agents in ceramic bodies to produce structures, having desirable strength and porosity, at temperatures below those required by the feldspathic fluxes is not new.[1] According to the Encyclopædia Britannica this idea had its inception not later than 1581, when a factory was established at Florence under the patronage of Francesco de Medici to produce white porcelain by introducing common glass into the body. The manufacture was continued at Pisa, and similar porcelain is attributed to Candiana near Padua. Another factory, founded at St. Cloud,

[1] Encyclopædia Britannica, vol. 18, 14th ed., p. 351, which refers to "Pottery and Porcelain, A Handbook for Collectors." Vol. III, E. Hannover (Ed. B. Lockham). A. B. Searle, Encyclopædia of Ceramic Industries, vol. 2, p. 377.

had grown to considerable size by the end of the seventeenth century. Searle states that porcelains, heated at low temperatures and made translucent by the aid of especially prepared glassy mixtures of frit were first produced at St. Cloud in 1727 and at Vincennes in 1740. These porcelains are now known as Pate Tendre or soft paste and are matured at temperatures above 1,000° C.[2] The same idea has been applied in more recent times to the manufacture of a type of ware named belleek, presumably after Belleek in County Fermanagh, Ireland.

The last few years have witnessed a renewed interest in the use of artificially prepared fluxes. With the development of methods for utilizing the electric current as a means for producing controlled temperatures, there naturally arises the question of the possibility of adapting this source of heat to the maturing of ceramic whiteware. It has been proven practicable to heat some classes of whiteware by passing the electric current through nonmetallic resistors. However, it would be an added economy and convenience if bodies of satisfactory qualities could be matured at, or below, 1,000° C. and consequently in furnaces of the metallic resistor type. The information contained in the literature [3] is lacking in specific data, particularly with reference to bodies produced at temperatures below 1,000° C. Moreover the field covered by the patent literature referred to is in general that of earthenware.

It was therefore the purpose of this investigation to determine the properties of a limited number of bodies having low absorptions and maturing below 1,000° C., and to obtain results serving as a guide to future industrial developments.

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[2] Temperatures are expressed in degrees Centigrade throughout this report.

[3] German Patent No. 182107 (Mar. 19, 1907); British Patents Nos. 253184 (June 9, 1926), 302519 (Dec. 20, 1928), 318188 (Aug. 30, 1929), and 323379 (Jan. 2, 1930); U. S. Patents Nos. 1749642 (Mar. 4, 1930), Re. 17656 (May 6, 1930) and 1819686 (Aug. 18, 1931). Also, P. F. Collins, J. Am. Cer. Soc., vol. 11 (9), p. 706, 1928; and vol. 15 (1), p. 17, 1932.

TABLE 1.—*Compositions of fluxes, given in molecular proportions*

Flux No.	Na_2O	MgO	SrO	ZnO	CaO	Al_2O_3	B_2O_3	Sb_2O_3	P_2O_5	SiO_2	ZrO_2	F	Remarks
	(RO=1.0)					(R_2O_3=1.3)				(RO_2=1.7)			
[1]1	1.0	------	------	------	------	--------	1.3	--------	------	1.7	------	------	Fuses readily, pours easily; glass colorless.
2	.8	0.2	------	------	------	--------	1.3	--------	------	1.7	------	------	Same as No. 1.
3	.6	.4	------	------	------	--------	1.3	--------	------	1.7	------	------	Fuses more readily than Nos. 1 and 2, pours easily; glass colorless.
4	.8	.2	------	------	------	0.1	1.2	--------	------	1.7	------	------	Tendency to foam during melting, melts and pours less readily than No. 1; glass clear light yellow (light blue when melted in platinum).
5	.8	.2	------	------	------	.2	1.1	--------	------	1.7	------	------	Tendency to foam greater than No. 4; otherwise similar.
6	.95	------	0.05	------	------	--------	1.3	--------	------	1.7	------	------	Fuses readily, pours easily; glass clear to very light yellow.
7	.95	------	.05	------	------	.2	1.1	--------	------	1.7	------	------	Tendency to foam, melts and pours similar to No. 5. Color clear to light yellow.
8	.9	------	.1	------	------	.2	1.1	--------	------	1.7	------	------	Melts and pours similar to No. 4; glass clear yellow (clear light blue when melted in platinum).
9	.8	------	------	0.2	------	--------	1.3	--------	------	1.7	------	------	Tendency to foam but melts and pours readily; glass colorless.
10	.5	------	------	.5	------	--------	1.3	--------	------	1.7	------	------	Melts and pours less readily than No. 9, otherwise similar.
11	.2	------	------	.8	------	--------	1.3	--------	------	1.7	------	------	Melts fairly readily to a white viscous glass; sample melted in platinum shows small amounts of doubly refracting crystals.
12	------	------	------	1.0	------	--------	1.3	--------	------	1.7	------	------	More difficult to melt but less opaque than No. 11. Too viscous to pour. Melt consisted of nearly pure glass of index 1.505.
13	------	------	------	1.0	------	.3	1.0	--------	------	1.7	------	------	More refractory than No. 12; no satisfactory melts obtained of either Nos. 12, 13, or 14.
14	------	------	------	1.0	------	--------	1.0	0.3	------	1.7	------	------	Did not fuse completely. Evidence of the reduction of zinc, also volatilization of ZnO or Sb_2O_3, or both.
15	.9	.05	.05	------	------	--------	1.3	--------	------	1.7	------	------	Fuses and pours readily; glass colorless.
16	.9	.05	.05	------	------	.2	1.1	--------	------	1.7	------	------	Same as No. 15, except for tendency to foam during melting.
17	.8	.2	------	------	------	--------	1.3	--------	------	1.2	0.5	------	Same as No. 15, except that sample melted in platinum was a clear light yellow.
18	1.0	------	------	------	------	--------	1.3	--------	------	1.2	.5	------	Same as No. 15, except amber in color; sample melted in platinum was clear greenish yellow.
19	.85	------	------	------	0.15	--------	1.3	--------	------	1.4	------	0.3	Less fusible than No. 15, but pours satisfactorily; glass clear light yellow.
20	1.0	------	------	------	------	.3	1.0	--------	------	1.4	------	.3	Tendency to foam, fuses fairly readily; glass clear light yellow.
21	.6	.4	------	------	------	.05	1.25	--------	------	1.4	------	.3	Melts and pours readily; glass clear amber.
22	.9	------	.1	------	------	.05	1.25	--------	------	1.4	------	.3	Foams during melting; fuses with difficulty; glass clear with bluish tinge.
23	.5	------	------	.5	------	.05	1.25	--------	------	1.4	------	.3	Fuses and pours satisfactorily although more viscous than Nos. 19 to 22, inclusive; glass clear with bluish tinge.
24	.8	------	------	------	.2	--------	1.2	--------	0.1	1.7	------	------	Very refractory and unsatisfactory as a frit; quenched mass composed principally of glass of index 1.515 to 1.522 and 4 to 5 per cent of very fine crystals.
25	1.0	------	------	------	------	.3	1.0	--------	------	1.7	------	------	Fuses fairly readily, pours easily, no foaming; glass clear with tinge of yellow.
26	1.0	------	------	------	------	.6	.7	--------	------	1.7	------	------	Considerable foaming during fusion, more refractory than No. 25 and does not pour readily; glass clear greenish yellow.
27	1.0	------	------	------	------	.8	.5	--------	------	1.7	------	------	Could not be melted satisfactorily.
28	1.0	------	------	------	------	.45	.85	--------	------	1.7	------	------	Tendency to foam, easier to fuse and pour than No. 26; glass clear with yellow tinge.

[1] Flux No. 1 is of the same composition as flux No. 24 in Auxiliary Fluxes in Ceramic Bodies, by P. F. Collins, J. Am. Cer. Soc. vol. 15 (1), p. 17, January, 1932.

II. MATERIALS

The following materials were used in preparing the artificial fluxes (Table 1): Borax, boracic acid, hydrous magnesium carbonate, zinc oxide, zirconium oxide, fluorspar, cryolite, antimony oxide, potters' flint, and kaolin from Georgia, all of commercial grade; also, sodium nitrate, strontium nitrate, strontium carbonate, and calcium phosphate, all of reagent quality.

The bodies (Table 2) were composed of two or more of the following materials in addition to one of the fluxes: Kaolin from Georgia, ball clay from Tennessee, feldspar containing approximately 50 per cent plagioclase feldspar, 40 per cent potash feldspar and 10 per cent quartz, and artificial "mullite" which was an electric furnace product containing approximately 65 per cent mullite, 12 per cent corundum, and 23 per cent glass. A commercial "soda-lime-silica" bottle glass, ground to pass a No. 100 sieve,[4] was substituted as the flux in one body having the type composition of the "BM Series."

All of the materials used in preparing the glaze (Table 2) were of commercial grade as regularly supplied to the pottery trade.

TABLE 2.—*Compositions of bodies and glaze used in the final series of tests*

A. BODIES[1]

	Series—			
	B	K	BM	KM
	Per cent	*Per cent*	*Per cent*	*Per cent*
Flux	25	25	25	25
Feldspar	35	35	35	35
Ball clay	40	20	35	17½
Kaolin	----	20	----	17½
Mullite	----	----	5	5

B. GLAZE

Fritt		Glaze[2]	
	Per cent		*Per cent*
Borax	10.3	Fritt	54.4
Boric acid	27.8	Whiting	4.8
White lead	24.3	White lead	16.5
Flint	37.6	Kaolin	24.3

[1] Body compositions are identified by a number followed by the series designation. Thus, 10 BM denotes a body of Series BM in which flux No. 10 was used.

[2] The coefficient of linear thermal expansion (calculated by means of factors in "The Influence of Chemical Composition on the Physical Properties of Glazes," by F. P. Hall, J. Am. Cer. Soc., vol. 13 (3), p.182, 1930) is 5.9×10^{-6}.

III. METHODS

The fluxes were melted in clay crucibles using a gas-fired pot furnace and, when molten, were poured into water, dried, and dry ground to pass a No. 100 sieve. The crucibles held from 1½ to 2 kg of the molten flux and a different crucible was used for each flux.

Relative solubility of the fluxes was determined on specimens obtained by dry sieving a portion of each flux through No. 200 and

[4] Sieves manufactured to meet the requirements of the United States Standard Sieve Series were used throughout this investigation.

No. 270 sieves, using 10 g of that portion which passed the No. 200 and was retained on the No. 270. The specimen was placed in a glass container, together with 200 ml of distilled water, agitated for 48 hours, the liquid removed by filtering, and the residue dried and weighed.

Thermal expansions and "softening temperatures" of the fluxes were determined by the interferometer method [5] on samples of flux which were remelted in platinum, cooled rapidly, and then annealed by heating to their annealing temperatures at an average rate of 2° per minute and holding them at these temperatures for one hour and cooling slowly with the furnace. The "softening temperature" was taken arbitrarily as that temperature at which contraction due to softening began to exceed the expansion due to temperature increase.

Indices of refraction of the fluxes were determined by the Becke method.[6]

The bodies were prepared by wet grinding the constituents in a ball mill for two hours, passing them over a magnetic separator and through a No. 100 sieve, and partly drying on plaster. This procedure, and the compositions used, were established after preliminary trials in which various compositions, modifications of wet and dry grinding, addition of flux before and after grinding, and filter pressing were tried.

Specimens for measuring transverse strength, linear shrinkage, and absorption were prepared by kneading the partly dried bodies and extruding them as rods one-half inch in diameter which were cut into 6-inch lengths. The rods were heated to about 750° C. overnight in an electrically heated furnace and matured the next day. Transverse strength is expressed as the modulus of rupture and was calculated from values obtained by breaking the rods across a 5-inch span. Percentage water absorption (which is proportional to porosity) was calculated from the weights of the specimens after having been dried at 115° ± 5° C. and after having been saturated by immersing in water and "autoclaving" for five hours at a steam pressure of 150 lbs./in.^{2}. Linear shrinkage of the rods was determined by measuring the distance, after the heating, between two gage marks which had been pressed 10 cm apart into the freshly extruded rods.

Additional extruded and dried rods were later reground, moistened with about 10 per cent water, passed through a No. 35 sieve, and used in making tile (mold $2\frac{7}{32}$ inches square) and plates (mold 3 inches in diameter) by the "dry-press" method. Total pressure used for both tile and plates was 12,000 pounds. The tile and plates were heated to about 750° C. overnight, in an electrically heated furnace, and matured the next day. Linear shrinkage, absorption, and relative resistance to abrasion of the dry pressed tile were determined on unglazed tile. Linear shrinkage was calculated from the dimensions of the steel mold and of the finished tile. Absorption was determined by the same me thod used for the extruded rods. Resistance to abrasion, a purely relative value expressed as the H_a value, was obtained by using the apparatus and method of calculation devised by D. W. Kessler [7] for wear tests of flooring materials. Glazed tile and glazed

[5] C. G. Peters and C. H. Cragoe, B. S. Sci. Paper No. 393.
[6] E. S. Larson, U. S. Geological Survey Bulletin No. 679, p. 14, 1921.
[7] A. S. T. M. Proc., vol. 28, Pt. II, p. 855, 1928.

plates were "autoclaved" in water for one and one-half hours at 150 lbs./in.^{2} steam pressure and the number of specimens crazed as a result of this treatment was noted. The "translucency" of a glazed plate was estimated by comparing the intensity of the light it transmitted with that transmitted by an arbitrarily chosen plate of medium "translucency," when such plates were placed over a 2½-inch diameter opening in a box which contained a 75-watt electric light, the filament of which was as near to the opening as possible. The glazed plates were between 3½ and 4 mm in thickness.

TABLE 3.—*Some physical properties of the fluxes*

Flux No.	Solubility		Softening temperature±10° C. Annealed [2]	Index of refraction	Coefficient of linear thermal expansion.[3] Annealed [2]	Flux No.	Solubility		Softening temperature±10° C. Annealed [2]	Index of refraction	Coefficient of linear thermal expansion.[3] Annealed [2]
	A [1]	B [1]					A [1]	B [1]			
	g	*g*	*° C.*		×10^{-6}		*g*	*g*	*°C.*		×10^{-6}
1	26	28	535	1.524	11.8	16	7	8	545	1.525	11.0
2	21	22	540	1.514	11.0	17	17	17	530	1.536	10.1
3	11	11	545	1.516	9.4	18	25	28	525	1.538	11.2
4	16	18	540	1.515	10.4	19	12	11	515	1.526	11.7
5	8	8	540	1.523	10.6	20	21	21	490	1.504	12.3
6	15	15	540	1.527	11.2	21	17	17	500	1.506	10.0
7	7	10	545	1.517	11.4	22	16	17	510	1.515	12.1
8	5	6	540	1.522	11.2	23	15	16	480	1.519	9.1
9	24	24	530	1.527	10.9	25	10	11	525	1.524	11.5
10	15	15	525	1.526	8.0	26	1	2	535	1.516	11.6
11	5	6	540	1.529	5.4	28	-------	2	545	1.515	11.1
15	14	13	535	1.524	10.7						

[1] A and B are duplicate determinations calculated on the basis of using 50 g of flux per liter of water.
[2] Fluxes Nos. 20, 21, 22, and 23 were annealed at 475° C., all others at 515° C.
[3] Average coefficient for the temperature range from room temperature to the lower critical temperature. For further information on the "critical temperatures" noted in studies of the thermal expansion of glass see the reference given in Footnote 5. Values to be multiplied by 10^{-6}.

IV. RESULTS

1. FLUXES

A brief description of the melting behavior of the fluxes and their appearance after quenching is given in Table 1. Flux No. 1 is of the same composition as flux No. 24 in the series investigated by Collins [8] and was selected as having a melting point and water solubility justifying its use as a basis on which to build the series of fluxes for this investigation. In outlining the series it is evident that no attempt was made to exhaust the field of possibilities.

Six of the twenty-eight fluxes (Nos. 3, 4, 7, 10, 11, and 28) were selected for intensive study. This choice was based on the observed melting behavior, the physical properties given in Table 3, and the unreported results obtained in a preliminary study. The compositions of the six selected fluxes as determined by calculations based on the batch materials, and by chemical analysis, are given in Table 4. The physical properties determined are presented in Table 5.

Although no specific tests were made of abrasives bonded with the fluxes investigated, it is believed, from observation, that at least some

[8] J. Am. Cer. Soc., vol. 15 (1), p. 17, January, 1932.

of the fluxes have possibilities as bonds for certain classes of manufactured abrasive wheels.

TABLE 4.—*Chemical composition* [1] *of fluxes Nos. 3, 4, 7, 10, 11, and 28*

Flux No. and method	SiO_2	Al_2O_3	CaO	MgO	Na_2O	B_2O_3	ZnO	SrO	Loss on ignition
	Per cent	*Per cent*	*Per cent*	*Per cent*	*Per cent*	*Per cent*	*Per cent*	*Per cent*	*Per cent*
3A [2]	37.6	0.42	0.09	5.1	16.7	41.0			0.31
3C [3]	41.5			6.5	15.1	36.8			
4A	38.8	3.9	.08	3.1	19.5	32.9			.22
4C	40.3	4.0		3.2	19.5	33.0			
7A	35.0	7.4		.13	24.1	32.3		1.8	.32
7C	38.8	7.7			22.4	29.1		1.9	
10A	37.8	.42	.05	.03	12.2	35.3	14.9		.23
10C	38.6				11.7	34.3	15.4		
11A	38.5	.53	.02	.02	4.8	33.7	23.6		.34
11C	37.8				4.6	33.5	24.1		
28A	36.6	17.4	.07	.12	23.6	22.1			.20
28C	38.0	17.0			23.0	22.0			

[1] The chemical analyses were made by J. F. Klekotka and are based on samples dried at 110° C.
[2] Compositions determined by chemical analysis (A).
[3] Calculated compositions based on batch materials (C).

2. BODIES

(a) PRELIMINARY STUDY

The results of this study are not presented in detail, but may be summarized briefly as follows:

Either a dry grinding of all of the constituents, or the addition of the flux after the other constituents of the body have been wet ground, produced coarse textured bodies.

Wet grinding four hours or more introduced so much dissolved flux into the slip that both the slip and the body were unworkable.

Sufficient flux was dissolved during two hours wet grinding to make filter pressing difficult.

Filter cakes contained sufficient water to carry flux to the surface, resulting in the formation of a hard, discolored "skin." (It is suggested that excess water could be removed from these bodies most efficiently by means of spray driers or drum driers.)

No method was found for preparing bodies which could be "jiggered" successfully. Such faulty specimens as were made contained sufficient water to carry dissolved flux to the surface during the drying.

Casting slips made with bodies containing fluxes 3 and 10 worked well on a laboratory scale and thin-walled pieces could be cast without difficulty. Fluxes Nos. 4, 7, 8, 11, and 28 were fairly satisfactory, while others, notably Nos. 1, 16, and 20, produced slips entirely unsuited for casting. However, rapid disintegration of molds was observed with all of the body slips because of the crystallization of dissolved flux carried into them by water from the slip. It was noted that pH values of the slips when removed from the ball mills could not be used as indicators of casting properties in so far as the particular bodies studied are concerned. Slips of bodies containing flux No. 10 showed the lowest pH values determined (approximately 6.0), while the slips containing flux No. 3 showed the highest (approxi-

mately 8.5). McDowell[9] states the pH value at which maximum dispersion is attained is different for each clay and for each electrolyte.

TABLE 5.—*Some physical properties of the fluxes used in the final series of tests as determined by calculation* [1]

Flux No.	Tensile strength	Modulus of elasticity	Coefficient of thermal expansion [2]	
			Calculated	Observed[3]
	kg/mm [2]	*kg/mm* [2]		
3	31.7	6,400	8.6	9.4
4	31.8	6,100	10.3	10.4
7	28.9	5,700	11.6	11.4
10	26.8	7,200	8.6	8.0
11	26.4	7,600	6.6	5.4
28	30.6	6,500	11.9	11.1

[1] Calculations based on the information contained in the reference given in the footnote to Table 2.
[2] Values to be multiplied by 10^{-6}.
[3] Values taken from Table 3.

No difficulty was experienced in making satisfactory specimens of tile and plates by the dry-press process.

Specimens made with 75 per cent flint and 25 per cent flux, when heated to 1,000° C., were translucent, bluish white in color, and of good texture, but the percentage tridymite developed was very high and the specimens were correspondingly sensitive to temperature change in the region of the α-β_1-β_2 tridymite transformations (117° and 163° C.).[10] The average coefficient of expansion for the temperature interval 100° to 200° C. was 41×10^{-6}.

Specimens made with various ratios of feldspar and flux formed highly translucent bodies, but the indicated maturing range did not exceed 10°.

A large majority of the trials indicated that additions of kaolin at the expense of ball clay darkened the color of the product.

Care must be observed in heating to allow ample time between 650° and 750° C. for complete combustion of organic matter before there is appreciable fusion of the flux and consequent sealing of the pores.

Holding at the maximum temperature for not less than one and one-half hours appears to permit the flux to distribute itself more uniformly throughout the body and produce a "smoother" appearing texture.

Specimens in which a commercial bottle glass had been used as a flux, and which had been heated at 990° ± 5° C., had an average absorption of 11.5 per cent. This indicates that commercial bottle glass would probably be unsuitable as a flux for bodies designed to mature below 1,000° C., and which are designed to have relatively low absorptions.

(b) PROPERTIES OF EXTRUDED BARS

The six fluxes selected for more intensive study were used in four series of bodies (Table 2) which have been labeled B, BM, K, and KM.

[9] J. Am. Cer. Soc., vol. 10 (4), p. 225, April, 1927.
[10] C. N. Fenner, Am. J. Sci., vol. 36, p. 331, 1913.

Although it was found that specimens could not be formed successfully from the plastic bodies by jiggering, it was thought advisable to obtain approximate results for their maturing range and other properties by means of conventional tests on extruded bars. Results were obtained for the B and BM series and are presented in Table 6. The results obtained give no ground for significant exceptions to the following statements: All of the rods developed maximum strength and shrinkage and minimum absorption when heated to 975° C. Heating to 1,000° C. was harmful. The minimum absorption was lower and the maturing range was shorter for bodies containing 5 per cent mullite than for bodies containing no mullite. Bodies made with a flux containing zinc (Nos. 60 and 61) showed a higher shrinkage and higher strength but not a lower absorption than the average for the series. The maturing range, whether or not it was desired to develop bodies of the lowest possible or of intermediate absorptions, did not exceed approximately 25° C.

The average values for modulus of rupture of the rods heated to 975° C. (Table 6) are very nearly equal to similar values which are found in the literature [11] for "semivitreous" bodies heated to cone 9 (about 1,250° C.) and which vary from 4,700 to 6,600 lbs./in.^{2}; but they are much lower than the values for vitreous bodies heated to cones 11 and 12 (about 1,285° and 1,310° C., respectively) which vary from 8,100 to 9,900 lbs./in.^{2}.

TABLE 6.—*Some properties of extruded bars (½ inch diameter by 6 inches long) heated for one-half hour at the temperatures indicated*

Body No.	900° C.			925° C.			950° C.			975° C.			1,000° C.		
	S	R	A	S	R	A	S	R	A	S	R	A	S	R	A
3B	1.4	3,000	8.0	2.1	3,400	6.5	2.2	3,500	5.0	3.0	4,000	3.5	3.1	3,500	2.0
4B	1.4	3,800	9.5	2.1	3,600	7.0	2.9	3,900	5.5	3.6	4,700	.5	3.1	4,000	4.5
7B	1.6	3,600	9.5	2.1	3,300	9.5	2.6	3,500	6.0	3.0	4,500	2.5	2.3	3,300	8.0
10B	1.7	3,200	10.0	3.1	3,700	7.5	3.8	4,500	5.0	4.9	4,300	3.5	3.3	3,500	7.0
11B	2.0	3,200	14.0	2.9	3,600	8.0	4.3	4,400	6.0	4.1	4,800	4.5	4.3	4,500	4.5
28B	1.4	3,300	12.5	2.5	3,800	9.0	3.3	4,400	7.0	3.5	4,600	6.0	3.5	4,300	5.0
3BM	1.6	3,600	9.0	2.0	4,100	6.0	2.2	3,900	5.0	3.6	4,600	.0	3.0	4,000	1.0
4BM	1.4	3,400	9.5	2.2	3,800	6.5	2.5	3,700	4.5	3.4	4,300	.5	2.5	3,400	4.0
7BM	1.6	3,800	8.0	2.0	3,800	6.0	2.5	4,000	3.5	2.8	4,600	.5	2.0	3,200	4.5
10BM	1.8	3,200	10.5	2.6	3,200	8.0	4.1	4,000	5.0	5.7	4,900	2.5	2.5	4,100	5.0
11BM	1.6	3,400	16.0	3.4	3,600	8.0	4.3	4,700	5.5	5.5	4,800	4.5	5.0	4,400	4.0
28BM	1.2	4,000	14.5	2.2	3,400	10.0	2.8	4,400	8.0	5.3	4,400	6.0	3.5	4,200	5.0

S=linear shrinkage, in per cent.
R=modulus of rupture, in pounds per square inch.
A=water absorption, percentage by weight.

(c) PROPERTIES OF DRY PRESSED TILE

(1) *Resistance to abrasion.*—The values obtained are given in Table 7. Apparently the compositions of the experimental bodies had no significant effect on their relative resistance to abrasion.

For comparison, three specimens of one brand of commercial white vitrified floor tile were tested and the average H_a value for nine determinations was 78. The following average H_a values are calculated from data in the report by Kessler:[12] Average of 45 marbles,

[11] E. E. Pressler and W. L. Shearer, B. S. Tech. Paper No. 310, 1926. R. F. Geller and A. S. Creamer, J. Am. Cer. Soc., vol. 14 (1), p. 30, January, 1931.
[12] See footnote 7, p. 477.

18; average of 15 limestones, 9; average of 14 slates, 8; average of 4 sandstones, 5.

(2) *Absorption.*—The absorption percentages reported in Table 7 are the lowest which it was possible to obtain without developing a vesicular or "overfired" structure. The group of bodies containing flux No. 3 has the lowest average absorption of the six groups differentiated by means of the flux. The BM series of bodies containing mullite but no kaolin has the lowest average absorption. Although there is little difference in the average absorption values given in Table 7 for the various series and groups, it should be remembered that those bodies containing flux No. 11 or No. 28 require a maturing temperature 30° C. higher than is required by the other bodies.

TABLE 7.—*Some properties of "dry pressed" tile*

Flux No.	Heat treatment		Resistance to abrasion;[1] series—				Absorption;[2] series—				Linear shrinkage;[3] series—			
	Maximum temperature, ±12° C.	Time at maximum temperature	B	BM	K	KM	B	BM	K	KM	B	BM	K	KM
	°C.	Hours	H_a value[4]				Per cent	Per cent	Per cent	Per cent	Per cent	Per cent	Per cent	Per cent
3	960	2–3	44	45	42	43	2.0	1.0	2.5	1.5	4.5	4.0	5.0	4.5
4	960	2–3	42	41	40	44	3.5	2.5	3.5	3.0	4.5	4.0	4.0	3.5
7	960	2–3	41	40	41	39	1.0	1.5	3.0	3.0	3.0	2.5	3.5	3.0
10	960	2–3	45	45	40	40	3.5	2.5	4.5	4.5	4.5	4.0	4.0	4.0
11	990	3	52	39	45	49	2.5	2.0	4.0	3.0	6.0	5.0	5.5	5.5
28	990	3	51	53	42	44	3.0	1.5	5.0	3.0	4.5	5.0	4.5	4.5

	Standard deviation		Maximum individual deviation
	Maximum	Minimum	
H_a value	7	1	10
Absorption, per cent	1.3	.3	2.5
Shrinkage, do	.8	.2	1.7

[1] Bodies 3, 4, 7, and 10, values are average of 6 specimens from 2 heatings. Bodies 11 and 28, values are average of 3 specimens from 1 heating.

[2] Bodies 3, 4, 7, and 10, values are average of 9 specimens from 3 heatings. Bodies 11 and 28, values are average of 6 specimens from 2 heatings.

[3] Number of specimens used same as for absorption. These values are the total linear shrinkage during drying and heating.

[4] Calculated by means of the formula $H_a=\frac{10\,(2{,}000+W_s)G}{2{,}000\,W_a}$ (see footnote 7, p. 477) where:

W_a=weight of material lost by abrasion.
W_s=original weight of specimen.
G=apparent density.

The absorptions of 180 specimens representing 17 brands of commercial tableware were determined. The lowest average absorption for specimens of any one brand was 0.2 per cent, the highest 9.5 per cent, and the average for all brands was 7.2 per cent. The deviations for the experimental bodies (Table 7) may be compared with the following deviations for the commercial bodies: The maximum standard deviation, in per cent, for the specimens of a single brand was 2.1, the minimum 0.2; the maximum deviation of an individual specimen from the mean of a single brand was 5.0 per cent.

(3) *Linear shrinkage.*—The values for total percentage linear shrinkage during drying and heating are given in Table 7. The low values for the bodies containing flux No. 7 are an interesting development in view of the fact that these bodies are also among the least absorbent.

(4) *Color.*—None of the bodies are white. Within each series the colors vary according to the flux, as follows: Flux No. 28 produced the darkest body, then Nos. 7, 4, 3, 10, and 11. When compared with color standards [13] the B and BM series may be described as varying from cartridge buff, Plate XXX 19″ Y-O-Yf, toward white while the K and KM series vary from tilluel buff, Plate XL 17‴ O-Yf, toward white. Before making the comparisons the eyes of the investigator were tested for sensitivity to color in the colorimetry section, and found to be normal.

(5) *Resistance to crazing.*—The relative resistance to crazing is indicated by the values in Table 8. The BM series of bodies appears to have a resistance appreciably higher than the others and it will be noted that this series (Table 7) has the lowest average absorption. The total number of crazed specimens in the three groups containing flux Nos. 3, 7, or 28 is significantly lower than in the other three. No explanation for their apparently higher resistance was found.

(6) *Thermal expansion.*—The coefficient of thermal expansion of each of the bodies of the B and BM series was calculated from tests on one specimen of each body. (Table 9.) Their coefficients do not differ significantly from those of 16 commercial whiteware bodies (one test on each body) which varied from 6.7 to 7.5×10^{-6}.

TABLE 8.—*Relative translucency and resistance to crazing*

Flux No.	Tile [1]					Plates [2]					Relative translucency average applies to each series
	Maximum temperature ±10° C.	Number of specimens crazed (3 tested); series—				Maximum temperature ±10° C.	Number of specimens crazed (2 tested); series—				
		B	BM	K	KM		B	BM	K	KM	
3	960	1	0	0	0	945	0	0	1	0	"Bright".
4	960	2	0	2	0	950	1	0	2	2	Do.
7	960	0	0	1	1	940	0	0	0	0	Medium.
10	960	3	0	3	3	955	0	0	2	1	Between 28 and 11.
11	990	0	1	3	2	970	1	0	2	1	Nearly opaque.
28	990	0	0	1	0	980	0	0	1	1	Below medium.

[1] Bisque heated 3 hours at the maximum temperature. Glaze matured 1 hour at 950°±5°.
[2] Bisque heated from ½ to 1½ hours at the maximum temperature. Glaze matured at 950°±5°—time 1 to 1½ hours.

(d) PROPERTIES OF GLAZED PLATES

(1) *Resistance to crazing.*—Values indicating relative resistance to crazing are given in Table 7. The resistance of the BM series was highest and the average resistance of the "ball clay bodies" (B and BM) was higher than that of the "kaolin bodies" (K and KM), which is in agreement with the results of the tests on glazed tile. Results of the tests on tile and plates may not be directly comparable because the former were glazed on one side only while the latter were completely coated.

[13] Robert Ridgway, Color Standards and Color Nomenclature, printed by A. Hoen & Co., Baltimore, Md.

(2) *Translucency.*—The values for translucency, which are purely relative, were determined for three specimens of each body. As a means for comparison, plates of several brands of domestic vitrified hotel china (thickness 4.3 to 5.1 mm) were tested also. Their translucency varied from the arbitrarily selected "medium" to "bright." The light transmitted by the least translucent specimens (containing flux No. 11) was approximately of the same intensity as that transmitted by a specimen of commercial earthenware of 4 per cent water absorption.

The translucency values for the experimental bodies are given in Table 8 and, as indicated, they varied according to the flux. Other variations in body composition did not affect the light transmission sufficiently to be detected by the test method. That the bodies containing flux No. 11 are least translucent is explained probably by the fact that the flux itself is nearly opaque. (Table 1.)

TABLE 9.—*Coefficients of thermal expansion of bodies of the B and BM series*[1]

Body No.	Coefficient of expansion[2]	Body No.	Coefficient of expansion[2]
3B	7.6	3BM	7.4
4B	7.9	4BM	7.8
7B	8.6	7BM	9.1
10B	7.3	10BM	7.5
11B	6.5	11BM	6.4
28B	8.4	28BM	8.7

[1] The temperature range is from room temperature to a temperature below the point at which the body shows a marked increase in rate of expansion (approximately from 25° to 400° C.). The temperature at which the rate of expansion increases is nearly the same as the lower critical temperature of the flux used.
[2] Values to be multiplied by 10^{-6}.

V. SUMMARY

The results obtained in this investigation indicate that the production of ceramic bodies having water absorptions lower than the average for commercial earthenware, and maturing below 1,000° C., would require careful supervision and control of all of the manufacturing processes.

It would probably be necessary to wet-grind the flux with the other ingredients of the body to obtain a sufficiently fine-grained and uniform product. This would be done at the cost of introducing dissolved flux in the "slip."

No difficulty was encountered in forming tile and plates approximately 2 inches square and 3 inches in diameter, respectively, by the dry-press process in a laboratory press.

The particular body compositions used could not be matured on what would usually be considered a short heating schedule, since it was necessary to allow sufficient time for all organic matter to oxidize and volatile products to escape before the flux softened sufficiently to seal the pores of the body.

Comparatively small variations in the chemical composition of the flux or of the body in which it is used may have a disproportionate effect on the properties of the product. For example, the use of kaolin in place of 50 per cent of the ball clay decreased the resistance to abrasion of 9 out of 12 bodies (Table 7); the average absorption of

the bodies containing kaolin was about 50 per cent higher than the average for the others (Table 7) and their resistance to crazing when glazed was considerably lower (Table 8). Also, the substitution of kaolin for ball clay produced bodies slightly darker in color. The results, therefore, indicate that it would be advantageous to use all ball clay, preferably "washed" and low in organic matter, rather than a mixture of ball clay and kaolin.

The introduction of 5 per cent of "mullite" at the expense of the clay appeared to have some desirable effects. (Tables 7 and 8.)

Within any one series of bodies the flux appeared to be the predominating factor in determining the relative color and translucency of bodies. (Table 8.) Effects on the other properties are not so pronounced.

It is believed that at least some of the fluxes have possibilities as bonds for certain classes of manufactured abrasive wheels.

VI. CONCLUSIONS

In general, it may be stated that ceramic bodies comparable in transverse breaking strength to earthenware, nearly white in color and varying in water absorption from 1 to 5 per cent, have been made in the laboratory by the dry-press process, of commercially available materials, and by maturing below 1,000° C. The bodies may be expected to resist mechanical abrasion better than nonceramic flooring materials, such as marble, and glazed specimens resisted crazing satisfactorily in laboratory tests. There are no data available on which to base conclusions regarding the cost of manufacture under commercial conditions.

Washington, May 9, 1932.